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(54) Title: EDIBLE PRE-FORMED FILM BARRIER MATERIALS

(57) Abstract

An edible pre-formed film for retarding moisture transfer among components of food having different vapor pressures, comprising a hydrophilic polymer layer and a lipid layer adhered to the hydrophilic polymer layer such that the film has a thickness in the range of 0.035 to 0.150 milimeters. The lipid layer has a concentration of at least approximately 0.8 milligrams of lipid per square centimeter of hydrophilic polymer layer. The exposed surface of the lipid layer is oriented toward the higher vapor pressure component of the food whereby when the film is placed between components of foof having different vapor pressures, the film can maintain the existing vapor pressure gradient for substantial periods of time, thereby retarding moisture transfer among the food components.

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# EDIBLE PRE-FORMED FILM BARRIER MATERIALS

## FIELD OF THE INVENTION

The present invention relates to edible pre-formed film barrier materials and methods to make edible film barrier materials which films can be used to stabilize vapor pressure gradients existing between individual components of a multicomponent food product separated by the edible film.

## BACKGROUND OF THE INVENTION

Deleterious changes in the quality of food products can occur with inadvertent alteration of the moisture content of the food product. The driving force for moisture transfer in food products is primarily due to moisture partial pressure gradients existing between the food product and the environment and/or between components of a multicomponent food product. The partial pressures involved can be expressed in terms of water activity, $a_w$ which equals the partial pressure of water vapor exerted by the food material divided by the vapor pressure of pure water at the same temperature. Prevention of moisture transfer between the food product and the environment can be accomplished by using a moisture impermeable packaging

material, either edible or inedible. Prevention of moisture exchange among individual components of a multicomponent food product is more difficult.

Edible food coatings have been used to retard moisture transfer within foods. However, the ability of such coatings to maintain vapor pressure gradients among components for extended periods has not been proven. The prior art is generally cognizant of the use of carbohydrate, protein, and lipid coatings to retard moisture transfer in foods. Lipids include hydrophobic compounds such as fats, oils, and waxes, and are especially effective in retarding moisture transfer in foods. However, when lipids such as waxes were applied to food products, the coatings were not continuous, but contained pinholes and cracks which provided routes for moisture transfer. Carbohydrate compounds are capable of forming continuous coatings, but these compounds are generally hydrophilic and hence less effective in retarding moisture transfer in foods. Therefore, in the past, lipids have been used in conjunction with carbohydrates in order to form a continuous coating to prevent moisture transfer in foods.

In U.S. Patent No. 2,909,435 to Watters, et al, a double layer coating was applied sequentially on foods such as raisins. The first layer consisted of a polysaccharide, upon which was applied a second layer of a melted wax composition. The layers were applied to the food by brushing, spraying, or dipping, and were dried after application. In U.S. Patent No. 3,323,922 to Durst, an aqueous solution was prepared from starch or a carboxymethyl cellulose, a plasticizer, and a lipid. The coating was then applied to a food product by brushing, dipping, or spraying, and the coating was dried after application on the food product.

U.S. patents number 3,471,303 and 3,471,304 to Hamdy, et al disclose two types of coating compositions having as one constituent a cellulose ether. The cellulose ether was plasticized by various fatty acid derivatives such as

(1) an ester of a linear polysaccharide having alpha-glucoside linkages and a fatty acid having between 8 to 26 carbon atoms, or (2) a glyceride containing a fatty acid having between 6 to 20 carbon atoms, together with a fatty acid metal salt wherein the fatty acid contains between 16 and 22 carbon atoms. The coating composition in the Hamdy patents could be extruded as a molten sheet over the food to be coated, and then dried.

U.S. patent number 3,997,674 to Ukai, et al discloses an aqueous solution used to coat foods such as fresh fruits. The coating solution contained a water soluble high polymer such as methyl cellulose and also hydrophobic substances such as waxes or oils. In Ukai, the food product was coated and then dried.

Drying a food coating after application on a food has several disadvantages. It is difficult to control the thickness of such coatings. Furthermore, drying coated foods adds time and hence cost to the manufacture of such foods. Therefore, it would be advantageous to provide off-the-shelf, pre-formed films which could effectively retard or prevent moisture transfer between components having different vapor pressures in a multicomponent food product.

## SUMMARY OF THE INVENTION

The present invention is summarized as an edible pre-formed and dried film for retarding moisture transfer among components of a multicomponent food product. The film includes a hydrophilic polymer layer selected from the group consisting of edible, film-forming carbohydrates and proteins and a lipid layer adhered to the hydrophilic polymer layer such that the film comprising the hydrophilic polymer layer and the lipid layer has a thickness in the range of 0.035 millimeters to 0.150 millimeters, the lipid layer having a concentration of at least approximately 0.8 milligrams of lipid per square centimeter of hydrophilic polymer layer. When the film is

placed between components of food having different vapor pressures, the lipid layer is oriented toward the food component possessing the greater water activity so that the film can maintain the existing vapor pressure gradient for substantial periods of time, thereby retarding moisture transfer among the food components.

It is an object of the present invention to provide an edible pre-formed and dried film which can retard moisture transfer among components of a multicomponent food product during extended storage times, and over changing temperatures and humidities, and yet which film can be absorbed in the food product during heating or other preparation of the food product or if not fully absorbed, will be unobjectionable during consumption of the food.

It is a further object of the present invention to provide a method for making edible pre-formed films capable of retarding moisture transfer among components of a multicomponent food product over extended storage times.

It is a still further object of the present invention to provide a food product and method to make a food product which includes a plurality of components having different vapor pressures together with edible films separating individual components, which films can maintain existing vapor pressure gradients for substantial periods of time, and thereby retard moisture transfer among the food components.

Other objects, advantages, and features of the present invention will become more apparent from the following detailed description when taken in conjunction with the accompanying drawings.

## DESCRIPTION OF THE PREFERRED EMBODIMENT

The present invention discloses an edible film for retarding moisture transfer among components of a multicomponent food product. The film includes a hydrophilic polymer layer, and a semi-solid, edible lipid layer adhered to the hydrophilic polymer layer such that

the film, comprising the hydrophilic polymer layer and the lipid layer, has a thickness in the range of 0.035 millimeters to 0.10 millimeters. The hydrophilic polymer layer can be formed from any edible, water soluble, film forming carbohydrate or protein. Suitable polymers would include, for example, starch, cellulose ethers such as carboxymethyl cellulose, hydroxypropyl methyl cellulose, and methyl cellulose, and also albumen. The lipid layer is comprised of semi-solid, edible lipids of plant or animal origin such as hydrogenated oils (e.g., palm oil and soybean oil), saturated fatty acids, and edible waxes (e.g., beeswax and paraffin wax). The lipid layer has a concentration of at least approximately 0.8 milligrams of lipid per square centimeter of hydrophilic polymer layer. The surface of the lipid layer is hydrophobic, whereby when the film is placed between components of food having different vapor pressure, the film can maintain the existing vapor pressure gradient for substantial periods of time, and thereby retard moisture transfer among the food components.

There are two methods to prepare an edible film according to the present invention. The first method is called the Layer method. In the first step of the Layer method, a film-forming solution is provided, consisting of a hydrophilic polymer and water. The film-forming solution is then spread on a plate, preferably to a depth of approximately 0.75 millimeters. The plate is then dried for a time sufficient to form a cohesive hydrophilic polymer layer on the plate. Finally, a lipid layer is applied to the hydrophilic polymer layer by brushing, dipping, or spraying; the lipid layer is then solidified. The lipid layer concentration should be at least approximately 4.0 milligrams of lipid per square centimeter of hydrophilic polymer layer. The dried film consisting of a hydrophilic polymer layer and a lipid layer should have a thickness in the range of 0.10 to 0.15 millimeters. The dried film is finally removed from the plate and positioned on the food material. Alternatively,

such films can be stored until the components of the food material are assembled. Preferably, alcohol such as ethanol is added to the film-forming solution to decrease drying time. The ideal alcohol to water ratio by volume is approximately 2 to 1. In addition, polyethylene glycol can be added to the film-forming solution to aid in removal of the dried films from the plates and to improve adhesion between the lipid and non-lipid layers. The ideal hydrophilic polymer to polyethylene glycol ratio by weight is approximately 9 to 1.

The second method for making an edible film according to the present invention is the Emulsion method. According to the Emulsion method, a film-forming solution is provided, consisting of a hydrophilic polymer, water, and a lipid. The solution is warmed, if necessary, to melt lipids having high melting points. The film-forming solution is then spread on a plate, preferably to a depth of approximately 0.75 millimeters. The film-forming solution on the plate is then dried for a time sufficient to form a cohesive edible barrier film having the thickness desired and a lipid concentration in the range of 0.8 to 1.0 milligrams lipid per square centimeters of hydrophilic polymer layer. A thickness in the range of 0.035 millimeters to 0.045 millimeters is preferred. After drying, the plate is cooled and the film is removed from the plate. The film can then be positioned on the food material, or stored until the food components are assembled. As in the Layer techique, alcohol and/or polyethylene glycol can also be added to the film forming solution in the ratios given above.

In both the Layer and Emulsion methods, the preferred hydrophilic polymer is a cellulose ether, specifically, hydroxypropyl methylcellulose. In both methods, the preferred lipids are selected from the group consisting of hydrogenated oils (specifically, hydrogenated soybean oil and hydrogenated palm oil), saturated fatty acids, and edible waxes. The preferred saturated fatty acids are stearic acid and a mixture of stearic and palmitic acids;

the ratio of stearic acid to palmitic acid in the mixture is not critical.

Temperature control is important in preparing a film by the Emulsion method. Emulsions containing fatty acids with high melting points require high temperatures to melt and disperse the fatty acid. However, high temperatures result in a low viscosity of the film-forming solution and poor control over film thickness during plating. Also, high temperatures must be avoided during plating, otherwise vaporization of the alcohol will occur resulting in bubbles in the solution and possibly pin holes in the dried film. However, the solution must be warm enough to prevent solidification of the fatty acids during plating. Within seconds after plating, the fatty acids orient at the emulsion surface such that the hydrophobic portion of substantially all of the fatty acids in the fatty acid layer is oriented away from the hydrophilic polymer layer.

Example 1 -- Layer Method

Nine grams of the cellulose ether hydroxypropyl methylcellulose were dissolved in 100 milliliters of 90°C distilled water. After the hydroxypropyl methylcellulose was completely dissolved, 200 milliliters of 95% ethanol was added. The solution was thoroughly mixed and then one gram of polyethylene glycol was dissolved in the solution. Air bubbles in the solution were removed by reducing the pressure over the solution.

One hundred milliliters of the film forming solution were added to a spreader for thin layer chromotography (TLC) and the solution was plated onto three 8 inch by 8 inch TLC glass plates at a thickness of 0.75 millimeters. The plates were dried in an oven at approximately 90°C for 15 minutes, at which time a cohesive cellulose ether layer formed. The plates were then cooled to room temperature and the hydroxypropyl methyl cellulose layers were removed and weighed. 3.3 grams of a lipid material was painted onto the surface of each 8 inch by 8 inch hydroxypropyl

methyl cellulose layer. The film thickness (hydroxypropyl methyl cellulose layer plus lipid layer) averaged 0.1 millimeters. The two layers were then reweighed. The concentration of lipid deposited was 8 milligrams lipid per square centimeters of hydroxypropyl methyl cellulose layer.

## Example 2 -- Emulsion Method

The film-forming solution was prepared by dissolving 9 grams of hydroxypropyl methylcellulose in 100 milliliters of 90°C distilled water. After the hydroxypropyl methylcellulose was completely in solution, 200 milliliters of 95% ethanol was added. Next, 1 gram of polyethylene glycol was added to the solution. Finally, 3.4 grams of stearic acid was added to the solution. The solution was warmed to between 70 - 75°C in order to melt the stearic acid.

One hundred milliliters of the film-forming solution was added to the TLC spreader and plated onto three 8 inch by 8 inch glass TLC plates at a thickness of 0.75 millimeters. The coated plates were then dried in an oven at approximately 90°C for 15 minutes, at which time a cohesive edible barrier film formed. After drying, the plates were cooled and the films were peeled from the plates. The films had an average thickness of 0.04 millimeters, and an average stearic acid concentration of 0.8 milligrams stearic acid per square centimeter of hydroxypropyl methyl cellulose layer.

A food prepared in accordance with the present invention could include a plurality of components having different vapor pressures, such as pizza or filled pie crusts. Preferably, an edible film prepared in accordance with the present invention would be pre-formed and then positioned so as to separate the individual components. Such edible films would include a hydrophilic polymer layer, and a lipid layer adhered to the hydrophilic polymer layer such that the film, comprising the

hydrophilic polymer layer and the lipid layer, has a thickness in the range of 0.035 millimeters to 0.150 milliliters. The lipid layer preferably has a concentration of at least approximately 0.8 milligrams lipid per square centimeter of hydrophilic polymer layer. The exposed surface of the lipid layer is hydrophobic and should be oriented towards the food component having a higher vapor pressure, such as pizza sauce or pie fillings. Therefore, the hydrophilic polymer layer would be oriented next to the food component having the lower vapor pressure, such as a pizza crust or pie crust. With this orientation, the film can maintain the existing food component vapor pressure gradient for substantial periods of time, thereby retarding moisture transfer from the component having a higher vapor pressure to the component having a lower vapor pressure. The film would be absorbed into the component layers upon heating or other cooking preparation in the range of 45 to 75°C during preparation of multicomponent foods such as a pizza or a filled pie crust.

It is to be understood that modification of the above-described edible film, method for making an edible film, food product, or method of making a food product, is possible within the spirit of the present invention and thus the present invention should not be limited to the above-described specification but should be interpreted in accordance with the following claims.

CLAIMS

What is claimed is:

1. An edible film for retarding moisture transfer among components of a multi-component food product, the film comprising:

(a) a hydrophilic polymer layer selected from the group consisting of edible, film-forming carbohydrates and proteins; and

(b) a lipid layer adhered to the hydrophilic polymer layer such that the film, comprising the hydrophilic polymer layer and lipid layer, has a thickness in the range of 0.035 millimeters to 0.150 millimeters, the lipid layer having a concentration of at least approximately 0.8 milligrams of lipid per square centimeter of hydrophilic polymer layer, wherein the exposed surface of the lipid layer is hydrophobic whereby when the film is placed between components of food having different vapor pressures, the film can maintain the existing vapor pressure gradient for substantial periods of time, thereby retarding moisture transfer among the food components.

2. The edible film of Claim 1, wherein the lipid which comprises the lipid layer is a semi-solid, edible lipid selected from the group consisting of hydrogenated oils, saturated fatty acids, and edible waxes.

3. The edible film of Claim 2, wherein the lipid is a saturated fatty acid having from 12 to 22 carbon atoms per molecule.

4. The edible film of Claim 3, wherein the saturated fatty acid has a concentration in the range of 0.80 to 1.0 milligrams fatty acid per square centimeter of hydrophilic polymer layer and the saturated fatty acid is selected from the group consisting of stearic acid, palmitic acid, and mixtures thereof.

5. The edible film of Claim 2, wherein the lipid is selected from the group consisting of hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax.

6. The edible film of Claim 1, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

7. The edible film of Claim 6, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

8. The edible film of Claim 2, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

9. The edible film of Claim 8, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

10. The edible film of Claim 3, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

11. The edible film of Claim 10, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

12. The edible film of Claim 4, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

13. The edible film of Claim 12, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl

cellulose, and hydroxypropyl methyl cellulose.

14. The edible film of Claim 5, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

15. The edible film of Claim 14, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

16. A method of preparing an edible film, comprising the steps of:

(a) providing a film-forming solution of water and a hydrophilic polymer selected from the group consisting of edible, film-forming carbohydrates and proteins;

(b) spreading the film-forming solution on a plate;

(c) drying the film-forming solution on the plate for a time sufficient to form a cohesive hydrophilic polymer layer on the plate; and

(d) applying a lipid layer onto the hydrophilic polymer layer so that the concentration of lipid on the hydrophilic polymer layer is at least approximately 4.0 milligrams lipid per square centimeter of hydrophilic polymer layer.

17. The method of Claim 16, wherein the lipid is a semi-solid, edible lipid selected from the group consisting of hydrogenated oils, saturated fatty acids, and edible waxes.

18. The method of Claim 17, wherein the lipid is a saturated fatty acid having from 12 - 22 carbon atoms per molecule.

19. The method of Claim 18, wherein the lipid is

selected from the group consisting of stearic acid, palmitic acid, stearic acid-palmitic acid mixtures, hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax.

20. The method of Claim 16, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

21. The method of Claim 20, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

22. The method of Claim 17, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

23. The method of Claim 22, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

24. The method of Claim 18, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

25. The method of Claim 24, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

26. The method of Claim 19, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

27. The method of Claim 26, wherein the hydrophilic polymer is a cellulose ether selected from the group

consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

28. A method for making an edible film, comprising the steps of:

(a) providing a film-forming solution of water, a lipid, and a hydrophilic polymer selected from the group consisting of edible, film-forming carbohydrates and proteins;

(b) spreading the film-forming solution on a plate; and

(c) drying the film-forming solution on the plate for a time sufficient to form a cohesive edible barrier film of the desired thickness having a lipid concentration in the range of 0.8 to 1.0 milligrams lipid per square centimeter of hydrophilic polymer layer.

29. The method of Claim 28, wherein the lipid is selected from the group of hydrogenated oils, saturated fatty acids, and edible waxes.

30. The method of Claim 29, wherein the lipid is a saturated fatty acid having from 12 to 22 carbon atoms per molecule.

31. The method of Claim 29, wherein the lipid is selected from the group consisting of stearic acid, palmitic acid, stearic acid-palmitic acid mixtures, hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax.

32. The method of Claim 28, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

33. The method of Claim 32, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose,

and hydroxypropyl methyl cellulose.

34. The method of Claim 29, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

35. The method of Claim 34, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

36. The method of Claim 30, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

37. The method of Claim 36, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

38. The method of Claim 31, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

39. The method of Claim 38, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

40. The method of Claim 16, wherein the film-forming solution further includes alcohol in a ratio by volume of 2 parts alcohol to 1 part water.

41. The method of Claim 16, wherein the film-forming solution further includes polyethylene glycol in a ratio by weight of 9 parts hydrophilic polymer to 1 part polyethylene glycol.

42. A food product comprising:

(a) a plurality of components having different vapor pressures; and

(b) an edible film separating the individual components, the film comprising a hydrophilic polymer layer selected from the group consisting of edible, film-forming carbohydrates and proteins, a lipid layer adhered to the hydrophilic polymer layer such that the film has a thickness in the range of 0.035 millimeters to 0.100 millimeters, the lipid layer having a concentration of at least approximately 0.45 milligrams lipid per square centimeter cellulose ether layer, wherein the surface of the lipid layer is hydrophobic and oriented towards the food component having a higher vapor pressure whereby when the film is in position between components of food having different vapor pressures, the film can maintain the existing vapor pressure gradient for substantial periods of time, thereby retarding moisture transfer among the food components.

43. The food product of Claim 42, wherein the lipid which comprises the lipid layer is a semi-solid, edible material selected from the group consisting of hydrogenated oils, saturated fatty acids, and edible waxes.

44. The food product of Claim 43, wherein the lipid is a saturated fatty acid having from 12 to 22 carbon atoms per molecule.

45. The method of Claim 43, wherein the lipid is selected from the group consisting of stearic acid, palmitic acid, stearic acid-palmitic acid mixtures, hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax..

46. The food product of Claim 42, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

47. The food product of Claim 46, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

48. The food product of Claim 43, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

49. The food product of Claim 48, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

50. The food product of Claim 44, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

51. The food product of Claim 50, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

52. The food product of Claim 45, wherein the hydrophilic polymer is selected from the group consisting of starcn, cellulose etners, and albumen.

53. The food product of Claim 52, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

54. The food product of Claim 46, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

55. The food product of Claim 54, wherein the hydrophilic polymer is a cellulose ether selected from the

group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

56. A method of preparing a food product from separate components which have different vapor pressures, comprising the steps of:

(a) positioning an edible film having the composition of Claim 1 such that the hydrophilic polymer layer of the edible film is adjacent to the lower vapor pressure component; and

(b) positioning the higher vapor pressure component such that the hydrophobic surface of the lipid layer is adjacent to the higher vapor pressure component whereby the edible film can maintain the vapor pressure gradient between the separate components for substantial periods of time, thereby retarding moisture transfer from the higher vapor pressure component to the lower vapor pressure component.

57. A method of preparing a food product from separate components which have different vapor pressures, comprising the steps of:

(a) positioning an edible film having the composition of Claim 2 such that the hydrophilic polymer layer of the edible film is adjacent to the lower vapor pressure component; and

(b) positioning the higher vapor pressure component such that the hydrophobic surface of the lipid layer is adjacent to the higher vapor pressure component whereby the edible film can maintain the vapor pressure gradient between the separate components for substantial periods of time, thereby retarding moisture transfer from the higher vapor pressure component to the lower vapor pressure component.

58. A method of preparing a food product from separate components which have different vapor pressures, comprising the steps of:

(a) positioning an edible film having the composition of Claim 6 such that the hydrophilic polymer layer of the edible film is adjacent to the lower vapor pressure component; and

(b) positioning the higher vapor pressure component such that the hydrophobic surface of the lipid layer is adjacent to the higher vapor pressure component and therefore the edible film can maintain the vapor pressure gradient between the separate components for substantial periods of time, thereby retarding moisture transfer from the higher vapor pressure component to the lower vapor pressure component.

59. A method of preparing a food product from separate components which have different vapor pressures, comprising the steps of:

(a) positioning an edible film having the composition of Claim 7 such that the hydrophilic polymer layer of the edible film is adjacent to the lower vapor pressure component; and

(b) positioning the higher vapor pressure such that the hydrophobic surface of the lipid layer is adjacent to the higher vapor pressure component and therefore the edible film can maintain the vapor pressure gradient between the separate components for substantial periods of time, thereby retarding moisture transfer from the higher vapor pressure component to the lower vapor pressure component.

60. The method of Claim 28, wherein the film-forming solution further includes alcohol in a ratio of 2 parts alcohol to 1 part water.

61. The method of Claim 28, wherein the film-forming solution further includes polyethylene glycol in a ratio by weight of 9 parts hydrophilic polymer to 1 part polyethylene glycol.

AMENDED CLAIMS
[received by the International Bureau on 05 November 1985 (05.11.85); original claims 1-61 replaced by new claims 1-67 (12 pages)]

1. Canceled.

2. (Amended) The edible film of Claim 62 wherein the lipid layer includes a semi-solid, edible lipid selected from the group consisting of hydrogenated oils, saturated fatty acids, and edible waxes.

3. (Amended) The edible film of Claim 62 wherein the lipid layer includes a saturated fatty acid having from 12 to 22 carbon atoms per molecule.

4. The edible film of Claim 3, wherein the saturated fatty acid has a concentration in the range of 0.80 to 1.0 milligrams fatty acid per square centimeter of hydrophilic polymer layer and the saturated fatty acid is selected from the group consisting of stearic acid, palmitic acid, and mixtures thereof.

5. The edible film of Claim 2, wherein the lipid is selected from the group consisting of hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax.

6. (Amended) The edible film of Claim 62 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

7. (Amended) The edible film of Claim 6 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl

cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

8. (Amended) The edible film of Claim 2 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

9. (Amended) The edible film of Claim 2 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

10. (Amended) The edible film of Claim 3 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

11. (Amended) The edible film of Claim 3 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

12. (Amended) The edible film of Claim 4 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

13. (Amended) The edible film of Claim 4 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

14. (Amended) The edible film of Claim 5 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

15. (Amended) The edible film of Claim 5 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

16. A method of preparing an edible film, comprising the steps of:

(a) providing a film-forming solution of water and a hydrophilic polymer selected from the group consisting of edible, film-forming carbohydrates and proteins;

(b) spreading the film-forming solution on a plate;

(c) drying the film-forming solution on the plate for a time sufficient to form a cohesive hydrophilic polymer layer on the plate; and

(d) applying a lipid layer onto the hydrophilic polymer layer so that the concentration of lipid on the hydrophilic polymer layer is at least approximately 4.0 milligrams lipid per square centimeter of hydrophilic polymer layer.

17. The method of Claim 16, wherein the lipid is a semi-solid, edible lipid selected from the group consisting of hydrogenated oils, saturated fatty acids, and edible waxes.

18. The method of Claim 17, wherein the lipid is a saturated fatty acid having from 12 - 22 carbon atoms per molecule.

19. The method of Claim 18, wherein the lipid is selected from the group consisting of stearic acid, palmitic acid, stearic acid-palmitic acid mixtures, hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax.

20. The method of Claim 16, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

21. The method of Claim 20, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

22. The method of Claim 17, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

23. The method of Claim 22, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

24. The method of Claim 18, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

25. The method of Claim 24, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

26. The method of Claim 19, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

27. The method of Claim 26, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

28. A method for making an edible film, comprising the steps of:

(a) providing a film-forming solution of water, a lipid, and a hydrophilic polymer selected from the group consisting of edible, film-forming carbohydrates and proteins;

(b) spreading the film-forming solution on a plate; and

(c) drying the film-forming solution on the plate for a time sufficient to form a cohesive edible barrier film of the desired thickness having a lipid concentration in the range of 0.8 to 1.0 milligrams lipid per square centimeter of hydrophilic polymer layer.

29. The method of Claim 28, wherein the lipid is selected from the group of hydrogenated oils, saturated fatty acids, and edible waxes.

30. The method of Claim 29, wherein the lipid is a saturated fatty acid having from 12 to 22 carbon atoms per molecule.

31. The method of Claim 29, wherein the lipid is selected from the group consisting of stearic acid, palmitic acid, stearic acid-palmitic acid mixtures, hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax.

32. The method of Claim 28, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

33. The method of Claim 32, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

34. The method of Claim 29, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

35. The method of Claim 34, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

36. The method of Claim 30, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

37. The method of Claim 36, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

38. The method of Claim 31, wherein the hydrophilic polymer is selected from the group consisting of starch, cellulose ethers, and albumen.

39. The method of Claim 38, wherein the hydrophilic polymer is a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

40. The method of Claim 16, wherein the film-forming solution further includes alcohol in a ratio by volume of 2 parts alcohol to 1 part water.

41. The method of Claim 16, wherein the film-forming solution further includes polyethylene glycol in a ratio by weight of 9 parts hydrophilic polymer to 1 part polyethylene glycol.

42. Canceled.

43. (Amended) The food product of Claim 63 wherein the lipid layer includes an edible lipid selected from the group consisting of hydrogenated oils, saturated fatty acids, and edible waxes.

44. (Amended) The food product of Claim 63 wherein the lipid layer includes a saturated fatty acid having from 12 to 22 carbon atoms per molecule.

45. (Amended) The food product of Claim 63 wherein the lipid layer includes a lipid selected from the group consisting of stearic acid, palmitic acid, stearic acid-palmitic acid mixtures, hydrogenated palm oil, hydrogenated soybean oil, beeswax, and paraffin wax.

46. (Amended) The food product of Claim 63 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

47. (Amended) The food product of Claim 46 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

48. (Amended) The food product of Claim 43 wherein the constituents of the hydrophilic polymer layer include

constituents selected from the group consisting of starch, cellulose ethers, and albumen.

49. (Amended) The food product of Claim 43 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

50. (Amended) The food product of Claim 44 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

51. (Amended) The food product of Claim 50 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

52. (Amended) The food product of Claim 45 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

53. (Amended) The food product of Claim 52 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

54. (Amended) The food product of Claim 46 wherein the constituents of the hydrophilic polymer layer include constituents selected from the group consisting of starch, cellulose ethers, and albumen.

55. (Amended) The food product of Claim 54 wherein the constituents of the hydrophilic polymer layer include a cellulose ether selected from the group consisting of methyl cellulose, carboxymethyl cellulose, and hydroxypropyl methyl cellulose.

56. Canceled.

57. Canceled.

58. Canceled.

59. Canceled.

60. The method of Claim 28, wherein the film-forming solution further includes alcohol in a ratio of 2 parts alcohol to 1 part water.

61. The method of Claim 28, wherein the film-forming solution further includes polyethylene glycol in a ratio by weight of 9 parts hydrophilic polymer to 1 part polyethylene glycol.

62. (New) An edible film for retarding water transfer between materials exhibiting differing water vapor pressures, the film comprising means for maintaining a substantially unbroken film, free of a supporting substrate, including:

(a) a hydrophilic polymer layer including constituents selected from the group consisting of edible, film-forming carbohydrates and proteins; and

(b) a lipid layer adhered to the hydrophilic polymer layer and having an exposed hydrophobic surface

whereby, when the film is first formed and then placed between materials exhibiting differing water vapor pressures with the hydrophobic surface of the lipid layer presented toward the

material having the higher water vapor pressure, the film tends to maintain the existing water vapor pressure gradient between the materials to retard water transfer between the materials.

63. (New) A food product comprising:

(a) a first component having a selected water vapor pressure and a second component having a water vapor pressure higher than that of the first component, so that a water vapor pressure gradient exists between the first and second components; and

(b) an edible film separating the first component from the second component, the film including:

a hydrophilic polymer layer having constituents selected from the group consisting of edible, film-forming carbohydrates and proteins, and

a lipid layer adhered to the hydrophilic polymer layer and having an exposed, hydrophobic surface, the exposed, hydrophobic surface of the lipid layer being oriented toward the second component,

whereby the film tends to maintain the water vapor pressure gradient between the first and second components, retarding water transfer between them.

64. (New) A method of preparing a food product including separate components, a first component having a selected water vapor pressure and a second component having a water vapor pressure higher than that of the first component so that a water vapor pressure gradient exists between them, the method comprising the step of positioning an edible film having the composition and structure of the film of Claim 62 with the hydrophilic polymer layer of the film presented toward the first component and the hydrophobic surface of the lipid layer presented toward the second component, whereby the edible film tends to maintain the water vapor pressure

gradient existing between the first and second components to retard the transfer of water from the second component to the first component.

65. (New) A method of preparing a food product including separate components, a first component having a selected water vapor pressure and a second component having a water vapor pressure higher than that of the first component so that a water vapor pressure gradient exists between them, the method comprising the step of positioning an edible film having the composition and structure of the film of Claim 2 with the hydrophilic polymer layer of the film presented toward the first component and the hydrophobic surface of the lipid layer presented toward the second component, whereby the edible film tends to maintain any water vapor pressure gradient existing between the first and second components to retard the transfer of water from the second component to the first component.

66. (New) A method of preparing a food product including separate components, a first component having a selected water vapor pressure and a second component having a water vapor pressure higher than that of the first component so that a water vapor pressure gradient exists between them, the method comprising the step of positioning an edible film having the composition and structure of the film of Claim 6 with the hydrophilic polymer layer of the film presented toward the first component and the hydrophobic surface of the lipid layer presented toward the second component, whereby the edible film tends to maintain any water vapor pressure gradient existing between the first and second components to retard the transfer of water from the second component to the first component.

67. (New) A method of preparing a food product including separate components, a first component having a selected water vapor pressure and a second component having a water vapor pressure higher than that of the first component so that a water vapor pressure gradient exists between them, the method comprising the step of positioning an edible film having the composition and structure of the film of Claim 7 with the hydrophilic polymer layer of the film presented toward the first component and the hydrophobic surface of the lipid layer presented toward the second component, whereby the edible film tends to maintain any water vapor pressure gradient existing between the first and second components to retard the transfer of water from the second component to the first component.

# INTERNATIONAL SEARCH REPORT

International Application No PCT/US85/01293

**I. CLASSIFICATION OF SUBJECT MATTER** (if several classification symbols apply, indicate all) [3]

According to International Patent Classification (IPC) or to both National Classification and IPC

INT. CL.[4] A23L 1/00

U.S. CL. 426/89, 92,93,94,103,138

**II. FIELDS SEARCHED**

Minimum Documentation Searched [4]

| Classification System | Classification Symbols |
|---|---|
| U.S. | 426/89,92,93,94,103,106,138,302,307,415,578 |

Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched [5]

**III. DOCUMENTS CONSIDERED TO BE RELEVANT** [14]

| Category * | Citation of Document, [16] with indication, where appropriate, of the relevant passages [17] | Relevant to Claim No. [18] |
|---|---|---|
| Y | US,A, 4,066,796, PUBLISHED 03 JANUARY 1978, McKEE. | 42-59 |
| Y | US,A, 4,401,681, PUBLISHED 30 AUGUST 1983, DAHLE. | 42-59 |
| Y | US,A, 4,293,572, PUBLISHED 06 OCTOBER 1981, SILVA. | 1-15, 42-59 |
| Y | US,A, 3,031,309, PUBLISHED 24 APRIL 1962, BOGNER. | 42-59 |
| A | US,A, 3,778,515, PUBLISHED 11 DECEMBER 1973, ASHLEY. | — |
| A | US,A, 2,836,498, PUBLISHED 27 MAY 1958, FENNEMA. | — |
| P | US,A, 4,515,819, PUBLISHED 07 MAY 1985, SHINRIKI. | — |
| A | US,A, 4,416,910, PUBLISHED 22 NOVEMBER 1983, HAYASHI ET AL. | — |
| A | US,A, 2,721,142, PUBLISHED 18 OCTOBER 1955, SHINN ET AL. | — |

* Special categories of cited documents: [15]

"A" document defining the general state of the art which is not considered to be of particular relevance

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

**IV. CERTIFICATION**

| Date of the Actual Completion of the International Search [2] | Date of Mailing of this International Search Report [2] |
|---|---|
| 11 SPETEMBER 1985 | 17 SEP 1985 |
| **International Searching Authority** [1] | **Signature of Authorized Officer** [20] |
| ISA/US | Steven L. Weinstein<br>STEVEN L. WEINSTEIN |

Form PCT/ISA/210 (second sheet) (October 1981)

International Application No. PCT/US85/01293

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)

| Category * | Citation of Document, [16] with indication, where appropriate, of the relevant passages [17] | Relevant to Claim No [18] |
|---|---|---|
| Y | US,A, 3,830,944, PUBLISHED 20 AUGUST 1974, DIMITRIADIS ET AL. | 42-59 |
| Y | US,A, 3,667,963, PUBLISHED 06 JUNE 1972, KATTER ET AL | 42-59 |
| Y | US,A, 3,997,674, PUBLISHED 14 DECEMBER 1976, UKAI ET AL. | 1-15, 42-59 |
| A | US,A, 3,471,303, PUBLISHED 07 OCTOBER 1969, HAMDY ET AL. | — |
| A | US,A, 3,471,304, PUBLISHED 07 OCTOBER 1969, HAMDY ET AL. | — |
| A | US,A, 2,909,435, PUBLISHED 20 OCTOBER 1959, WATTERS ET AL. | — |
| A | US,A, 3,323,922, PUBLISHED 06 JUNE 1967, DURST. | — |
| P | US,A, 4,513,019, PUBLISHED 23, APRIL 1985, BRANCQ ET AL. | — |
| A | US,A, 3,388,085, PUBLISHED 11 JUNE 1968, LEVKOFF ET AL. | — |
| Y | US,A, 3,526,515, PUBLISHED 01 SEPTEMBER 1970, WERBIN ET AL. | 1-15, 42-59 |
| A | US,A, 3,615,647, PUBLISHED 26 OCTOBER 1971, KASSENS. | — |
| A | FR,A, 2,087,185 PUBLISHED 31 DECEMBER 1971, ROUSSELOT KUHLMANN. | — |
| A | JP,A, 51-91359, PUBLISHED 07 OCTOBER 1976, SUMITOMO BAKELITE. | — |
| A | JP,A, 52-12943, PUBLISHED 31 JANUARY 1977, SUMITOMO BAKELITE. | — |
| A | JP,B, 53-15100, PUBLISHED 22 MAY 1978, ASAHI CHEMICAL INDKK. | — |
| A | JP,A, 53-34939, PUBLISHED 31 MARCH 1978, KAWASHIMA. | — |
| A | GB,A, 2,105,168, 23 MARCH 1983, HOLT. | — |
| Y | N, CEREAL SCIENCE TODAY, PUBLISHED OCTOBER 1965, VOLUME 10, NUMBER 9, PAGES 508 AND 510. | 1-15, 42-59 |

International Application No. PCT/US85/01293

**FURTHER INFORMATION CONTINUED FROM THE SECOND SHEET**

| | | |
|---|---|---|
| A | N, INDUSTRIAL AND ENGINEERING CHEMISTRY, PUBLISHED OCTOBER 1945, PAGES 943-947 | — |
| A | N, INDUSTRIAL AND ENGINEERING CHEMISTRY, PUBLISHED APRIL 1951, PAGES 911-919. | — |
| A | N, MODERN PACKAGING, PUBLISHED SEPTEMBER 1948, PAGES 157 and 158. | — |

**V.☐ OBSERVATIONS WHERE CERTAIN CLAIMS WERE FOUND UNSEARCHABLE [10]**

This international search report has not been established in respect of certain claims under Article 17(2) (a) for the following reasons:

1.☐ Claim numbers ______, because they relate to subject matter [12] not required to be searched by this Authority, namely:

2.☐ Claim numbers ______, because they relate to parts of the international application that do not comply with the prescribed requirements to such an extent that no meaningful international search can be carried out [13], specifically:

**VI.☒ OBSERVATIONS WHERE UNITY OF INVENTION IS LACKING [11]**

This International Searching Authority found multiple inventions in this international application as follows:

I. Edible film, A composite food product and Method of Preparing Composite Food product: Claims 1-15, 42-59.

II. Method of Making the edible film by the layer method: claims 16-27, 40, 41.

III. Method of making the edible film by the emulsion method: cl. 28-39, 60,61.

1.☐ As all required additional search fees were timely paid by the applicant, this international search report covers all searchable claims of the international application.

2.☐ As only some of the required additional search fees were timely paid by the applicant, this international search report covers only those claims of the international application for which fees were paid, specifically claims:

3.☒ No required additional search fees were timely paid by the applicant. Consequently, this international search report is restricted to the invention first mentioned in the claims; it is covered by claim numbers:

4.☐ As all searchable claims could be searched without effort justifying an additional fee, the International Searching Authority did not invite payment of any additional fee.

Remark on Protest

☐ The additional search fees were accompanied by applicant's protest.

☐ No protest accompanied the payment of additional search fees.